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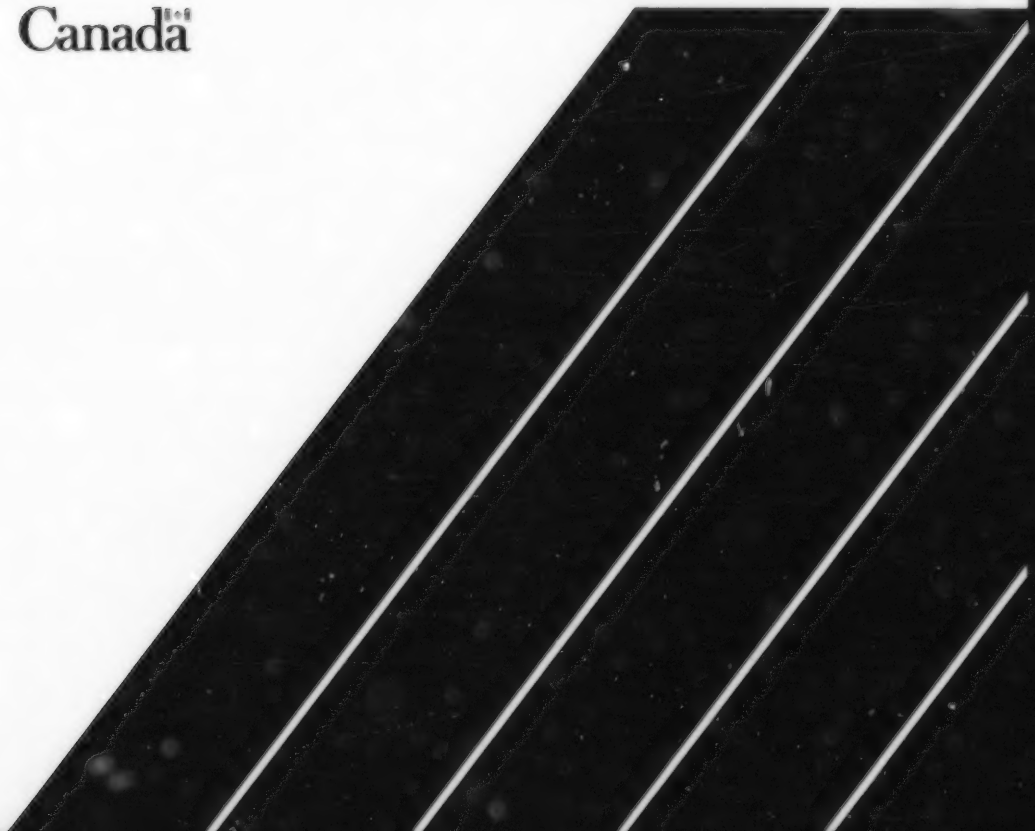
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MINIMUM IGNITION TEMPERATURES OF DUST LAYERS USING THE SETCHKIN APPARATUS

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**MINIMUM IGNITION TEMPERATURES OF DUST LAYERS
USING THE SETCHKIN APPARATUS**

by

K.J. Mintz*

ABSTRACT

The Setchkin flammability apparatus, designed for measuring the minimum ignition temperature (MIT) of plastics, was evaluated for use for measuring the MIT of dusts. It is shown that the apparatus can obtain reliable values of MIT with an estimated accuracy of $\pm 10^\circ\text{C}$. Pittsburgh standard coal dust and lycopodium dust were used as "standard" dusts. It is shown that there are actually two different MIT's; one for non-flaming decomposition (the beginning of an exothermic reaction) and the other for flaming decomposition. The latter does not appear to have been previously reported in the literature. Both MIT's have a strong dependence on sample size; some evidence points to sample mass being the more correct independent variable (for tests in the Setchkin apparatus) rather than layer thickness (which may be applicable for tests using a hot-plate apparatus).

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TEMPÉRATURES D'INFLAMMATION MINIMALES DES COUCHES DE POUSSIÈRE EN UTILISANT L'APPAREIL SETCHKIN

par

K.J. Mintz*

RÉSUMÉ

L'appareil de mesure du point d'inflammation Setchkin, conçu pour mesurer la température minimale d'inflammation (TMI) des plastiques, a été évalué en fonction de son utilisation pour la mesure de la TMI des poussières. Il semble que cet appareil peut donner des valeurs fiables de TMI avec une précision approximative de $\pm 10^{\circ}\text{C}$. La poussière du charbon étalon Pittsburgh ainsi que la poudre de lycopode ont été utilisées comme poussières de "référence". Les résultats indiquent qu'il y a deux différents TMI; une pour la décomposition sans flamme (le début d'une réaction exothermique) et l'autre pour une décomposition avec flamme. Cette dernière ne semble pas avoir été mentionnée antérieurement dans la documentation scientifique. Les deux TMI dépendent du format des échantillons. Certains éléments indiquent que la masse de l'échantillon est une variable indépendante plus exacte (pour les essais avec l'appareil Setchkin) que l'épaisseur de la couche de poussière (laquelle peut être utilisée dans les essais avec l'appareil à plaque chauffante).

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INTRODUCTION

The ignition of dust layers on hot surfaces is a potential hazard in many industries, because it can result in a major fire, or even an explosion (1). It is therefore important to be able to measure the maximum temperature that a dust can be exposed to without any combustion. In the laboratory, the parameter that is measured is the *minimum ignition temperature* (MIT). Note that the MIT of dust layers is **not** the same as the MIT of suspended dust, which is commonly measured by the Godbert-Greenwald Furnace (1).

There are two general ways of measuring the MIT of dust layers. One is by putting a sample on a hot-plate; in this case, only the bottom surface is exposed to the high temperature; the rest of the sample is exposed (more-or-less) to the ambient temperature. The International Electrotechnical Commission (Technical Committee 31H) has drafted a standard in this area, for use with electrical equipment (2). A similar procedure is in use on a recommended basis in the USA (3). The second method is the furnace method, in which the sample is surrounded by a uniform temperature, which has been used routinely by the U.S. Bureau of Mines (4). The values of the MIT are, in general, significantly lower using the second method. For the majority of industrial applications, the first method probably provides a closer approximation to reality. For some industrial applications, however, the second method is more applicable.

In both methods, the MIT is a function of the thickness of the layer: the thicker the layer, the lower the MIT (i.e., the more hazardous is the situation). Standard thicknesses used are 5 and 12.5 mm.

The present report deals with the second method, because it provides the more conservative values. In the future, an apparatus for the first method will be built and commissioned.

At least two apparatuses have been used for the furnace method (5): a modified version of the Godbert-Greenwald furnace and the Setchkin furnace. Both were available in this laboratory; the latter was chosen for this work, mainly because of convenience.

The purpose of the present work is to develop a method for routine use of the apparatus for measuring the MIT of dust layers and to study the effects of the various parameters on the results. Pittsburgh standard coal dust and lycopodium are widely used as standards; both of them were used in this study for this purpose.

EQUIPMENT

The Setchkin apparatus is described in ASTM Standard D1979 (6). The furnace consists of an inner ceramic tube of inside diameter 76 mm and length about 220 mm, inside is another ceramic tube, containing the heating unit. The unit is placed so that the tubes are vertical; air flows down the outer tube and up the inner tube. The top is covered by a split disk made of pyrex glass, which has a 25 mm diameter opening. A diagram of the apparatus is shown in Fig.1. The Standard is actually for measuring the MIT of plastics, not dusts; therefore, the sample holder specified is not suitable.

The unit used here was made by Custom Scientific instruments, Inc., Cedar Knolls, N.J. and consists of the furnace itself plus a control unit. The latter contains a pump and flowmeter for the supply of compressed air for the furnace, heating controls and a data logger. The latter is pre-programmed (not user-adjustable) so that it prints out temperatures from 5 K-type thermocouples every minute and charts the temperatures every 10 s. One thermocouple, used for control, is installed in the outer tube.

In order to see events occurring in the sample, a mirror holder was built and attached to a metal support positioned appropriately.

Two thermocouples are required for the tests (in addition to the control thermocouple): one positioned below the sample, which measures the furnace temperature, and the other, immersed in the sample. The thermocouples used were sealed and grounded type, 3.2 mm diameter. Exposed-end thermocouples were tried in preliminary trials, but they did not survive the corrosive gases generated during the tests.

The sample container used was a ceramic crucible (Coors model 60133), which has a diameter of 32 mm and a height of 20 mm. It fits into a stainless steel ring of 35 mm diameter, which was welded onto a stainless steel rod 190 mm long, with a hook on the end to fit over a support rod 30 mm above the top of the furnace.

CALIBRATIONS OF THE FURNACE

Before and after use, the thermocouples were checked using a temperature calibrator over the range 30 and 700°C. The maximum error in each thermocouple was 2°C.

According to ASTM D1929, the test must be carried out at different air flow rates, 25, 51 and 100 mm/s (5, 10 and 20 ft/min) in order to obtain the lowest MIT. The velocity of air at any temperature is set by using Fig. 2 in the standard to find the appropriate value of the volume flow rate at 23°C and then setting that value on the flowmeter on the control box. The flow rate used by the U.S. Bureau of Mines is somewhat smaller, only 8 mm/s. In order to evaluate the method properly, it was decided to cover the range from 0 - 100 mm/s in the present study.

First, the temperature in the furnace was measured, as a function of set point and air velocity. The results are shown in Fig. 2. The closed circles (connected by dashed lines) represent data taken at the position where the temperature of the sample is normally measured; the open circles (connected by solid lines) represent data taken where the furnace temperature is normally measured, about 5 cm below the "sample" thermocouple. The 10 SCFH corresponds to between 7 and 11 ft/min, depending on the temperature, which is in the mid-range of the velocity range specified in the standard. As can be seen, the "sample" thermocouple registers a slightly smaller value than the "furnace" thermocouple, that is, the temperature in the furnace is not quite uniform, but decreases from the bottom to the top. The difference, however, is not great enough to have a significant effect on the accuracy of the MIT. The difference in temperature between no flow and 10 SCFH is, however, definitely significant. The latter corresponds quite closely to the set temperature; the former is significantly lower. This is probably due to loss of heat from the inner tube unless a constant flow of air enters from the heated outer tube. These results do not imply that tests cannot be carried out at zero flow rate, but do indicate that this graph should be used to select the appropriate set temperature for the desired test.

The effect of placing the crucible in the furnace was determined, with the results shown in Fig. 3. The open symbols (connected by solid lines) represent data from the thermocouple used for measuring the furnace temperature; the closed circles (connected by dashed lines) represent data from the thermocouple located inside the crucible. As for Fig. 2, the furnace temperature increases relative to the set temperature as the flow rate increases. The difference between the set and furnace temperatures is about the same with and without the crucible at moderate flow rates, but is significantly reduced with the crucible at no flow. The reason is probably the trapping of warm air under the crucible. At all flow rates, the difference between the furnace and sample temperatures is greater when the crucible is in place. In other words, the temperature distribution is less uniform

due to the presence of the crucible. Presumably, the volume inside the crucible does not equilibrate with the air surrounding it because of lack of mixing. However, during an actual test, the thermocouple is immersed in the sample rather than air, and thus there is an opportunity to equilibrate by means of conduction as well as convection. Therefore, the difference in temperature between the furnace and sample may be less than that indicated from Fig. 3.

The MIT is usually determined in two stages. An approximate value is found by placing the crucible with the sample into the furnace at ambient temperature, heating up the furnace and noting the inflection point on the sample temperature-time plot. A more exact value is found by placing the sample into the furnace which has already been heated to the desired temperature (the initial guess being the temperature found in the first stage), and again observing the sample temperature-time trace. If an inflection is observed, then the furnace temperature is above the MIT; if no inflection is observed, then it is below the MIT. The furnace temperature is varied until the MIT is bracketed within the desired precision.

The data in this section show that the set temperature cannot be used for the constant temperature tests (second stage of the MIT determination), otherwise the MIT will be significantly overestimated when low flow rates are used and underestimated when high flow rates are used. It is necessary to use the furnace temperature for the determination of the MIT; the desired temperature to be set on the control box can be determined from the curves (solid lines) of Fig. 3.

For the constant temperature tests, the furnace temperature will decrease when the crucible with the sample is placed into the furnace, primarily because it is necessary to remove half the top of the furnace which results in a loss of heat. The furnace temperature stabilizes again in about 10 - 20 minutes, but not necessarily to the original temperature, because of the effects of the crucible noted above. Thus, true constant temperature tests cannot be actually achieved. The best approximation to the temperature that the sample is exposed to is to use the furnace temperature after the crucible has been inserted and the furnace has stabilized again. This is only required for zero flow rate tests. Otherwise, the actual "furnace" temperature measured just prior to inserting the sample will provide sufficient accuracy.

From the above calibrations, the most accurate data should be from tests carried out at moderate flow rates. Even so, the accuracy of the MIT should not be considered better than $\pm 10^{\circ}\text{C}$.

PITTSBURGH STANDARD COAL DUST

Effect of Flow rate

Tests were carried out using 2.0 g of Pittsburgh dust in the crucible, which makes a layer 12 mm deep. The results are shown in the following table:

Table 1. Pittsburgh Standard Coal Dust: Effect of Flow Rate on MIT

Flow rate (SCFH)	T _{set} (°C)	T _{furnace} (°C)	T _{max.} (°C)	T _{inflection} (°C)
0	250	209	262	230
	240	200	263	220
	235	192	244	220
	230	186	197	none
5	250	220	298	250
	210	192	250	220
	200	189	203	none
10	250	225	286	250
	230	210	294	230
	210	199	252	220
	200	190	242	220
	190	185	190	none

From the above table and using the assumptions in the previous section, the MIT at zero flow rate is between 186–192°C, at 5 SCFH 189–192°C, and at 10 SCFH 185–190°C. Hence, the MIT is not a function of the flow rate; its value is 190°C for the particular sample size used. Furthermore, it can be seen that there is a sharp break between ignition and non-ignition: ignition produces a significant temperature rise, with the T_{maximum} at least 50°C higher than the T_{furnace}; non-ignition produces a very small temperature rise. The inflection point is the temperature at which the slope of

the curve makes a sharp change; for T_{furnace} not too much higher than the MIT, $T_{\text{inflection}}$ is 220°C and is independent of flow rate.

Effect of Sample Size

The MIT of Pittsburgh coal dust was determined as a function of sample size, as shown in Table 2 (p.7). The sample size can be expressed either as layer thickness or as mass. Table 2 gives the highest T_{furnace} that did not produce ignition and the lowest T_{furnace} that resulted in an ignition. The inflection due to ignition becomes less sharp as the sample size is decreased, as shown in the column labelled $T_{\text{max}} - T_{\text{furnace}}$.

The MIT's from Table 2 are plotted as a function of sample layer thickness in Fig. 4 and of sample mass in Fig. 5. Both curves (cubic spline fit) are smooth, and show the characteristic of the MIT of dust layers: as the dust layer becomes smaller, the MIT rises sharply. The curve in Fig. 5 appears to have reached an asymptotic value of 175°C at larger sample masses. On the other hand, on the basis of Fig. 4, the asymptotic value of the MIT cannot be estimated, but it is clearly much lower. Which is more meaningful? A few additional tests were carried out in a quartz crucible of a narrower shape than the porcelain crucible used normally. In this case, 18 mm thickness corresponded to only 3.1 g. The MIT obtained was between 180 and 190°C . This value corresponds much closer to the curve in Fig. 5 than the one in Fig. 4.

The MIT obtained in a heated enclosure depends predominantly on the size of the deposit, whereas the MIT obtained on a hot-plate depends predominantly on the thickness of the layer. The literature values for 12.7 mm thick layers of Pittsburgh coal dust are $170 - 175^{\circ}\text{C}$ using the Godbert-Greenwald furnace. For that apparatus, a cylinder of 1 inch diameter was used to contain the sample. A 12.7 mm thick layer in that corresponds to a mass of about 8 g. Our data are in good agreement with those in the literature, if the comparison were based on mass, but not good if the comparison were made on layer thickness.

The most recent literature value for the MIT of a 12.7 mm thick layer of Pittsburgh coal dust, using the hot-plate method is 240°C , (3), though earlier tests had yielded 170°C (7). Palmer (1) has noted that enclosure tests should give lower values than hot-plate tests; hence, the former value is more likely than the latter.

Table 2. Pittsburgh Standard Coal Dust: Effect of Sample Size

Mass (g)	Depth (mm)	T_{furnace} (°C)	$T_{\text{max}} - T_{\text{furnace}}$ (°C)	$T_{\text{inflection}}$ (°C)
.3	3.2	243	13	244
		224	0	none
.5	4.3	237	32	230
		228	27	225
		216	4	none
1.0	7.6	219	37	230
		206	44	225
		199	6	none
2.0	12	209	53	250
		200	63	220
		192	52	220
		186	11	none
5.0	18	180	85	210
		175	81	205
		173	9	none
		164	3	none

MIT - Flaming Combustion

The MIT's determined in the previous sections represent temperatures at which more heat is produced by the sample than is absorbed from the environment. These MIT's are the ones quoted in the literature. But there is another type of MIT, in which the sample bursts into flame. The writer could find no record of these in the literature. They can be important to explain the occurrence of a fire observed by the dust, and to be able to define hazards of the dust more accurately.

The determination of flaming combustion MIT's is similar to the other MIT's except that the test is stopped when a flame is observed (in which case the furnace temperature is above the MIT), or the sample has decomposed quietly without flaming (in which case the furnace temperature is below the MIT). The values of MIT were determined as a function of sample size (see Fig. 6). Tests were also carried out with 0.1 g of Pittsburgh coal; even as high as 670°C, no flaming combustion occurred. The same type of variation with sample size occurred as with non-flaming combustion; the MIT appears to approach an asymptotic value at large sample sizes.

LYCOPODIUM

Non-flaming Combustion

The MIT of lycopodium was measured as a function of sample size, similar to Pittsburgh coal. The results are shown as a function of layer thickness in Fig. 7 and of sample mass in Fig. 8. (Cubic spline fits were used.) As for Pittsburgh coal, the curves appear to be close to an asymptotic value at the largest sample sizes. Note, however, the difference in the abscissae: lycopodium stabilizes at a smaller sample size than Pittsburgh coal.

The MIT for a 13 mm thick layer of lycopodium is about 185°C, compared to the value of 190°C determined by Miron and Lazzara in the Godbert-Greenwald furnace and 240°C in the hotplate test (3). The value of 310°C usually quoted for lycopodium appears to be in serious error.

Flaming Combustion

The results of the flaming combustion tests on lycopodium are shown in Fig. 9. The variation of MIT with sample size is rather small. The boundary between flaming and non-flaming was quite sharp. Flaming occurred, usually accompanied by a distinct "pop", about 1 - 4 minutes after the sample was placed in the furnace; the longest times were for temperatures close to the MIT.

DISCUSSION

This report has shown the existence of two modes of ignition of dust layers: one that occurs at lower temperatures in which an exothermic reaction occurs but no flames are generated, the other occurring at higher temperatures, in which flames are produced. Based on limited data, the latter appear to occur in the same range of temperatures as ignition of a dust cloud. This phenomenon of two modes of ignition does not appear in the literature.

The question can be raised as to the dependence of the $MIT_{\text{flaming combustion}}$ on the apparatus. Ignition occurs because the temperature is such that the combustible gases generated from the decomposition are generated at a sufficient rate that their concentration is above the MEC for auto-ignition. Hence, for a given material, the MIT_{flam} might be expected to be a function of the apparatus size, flow rate of air passing through the furnace, as well as sample size. Sample size has two effects: the quantity of gas generated per unit time and the ability to retain heat inside the sample. Only the latter is relevant for $MIT_{\text{non-flam}}$. Hence, it might be thought that MIT_{flam} would be less of an intrinsic property of a material than $MIT_{\text{non-flam}}$. On the other hand, it should be recognized that typically the rate of chemical reaction doubles every 10°C , and so the effect of temperature would predominate over the other effects. The variation of MIT_{flam} with sample size was found in this study to be in fact less than the variation of $MIT_{\text{non-flam}}$, thus indicating that the former parameter is at least as intrinsic a quantity as the latter parameter.

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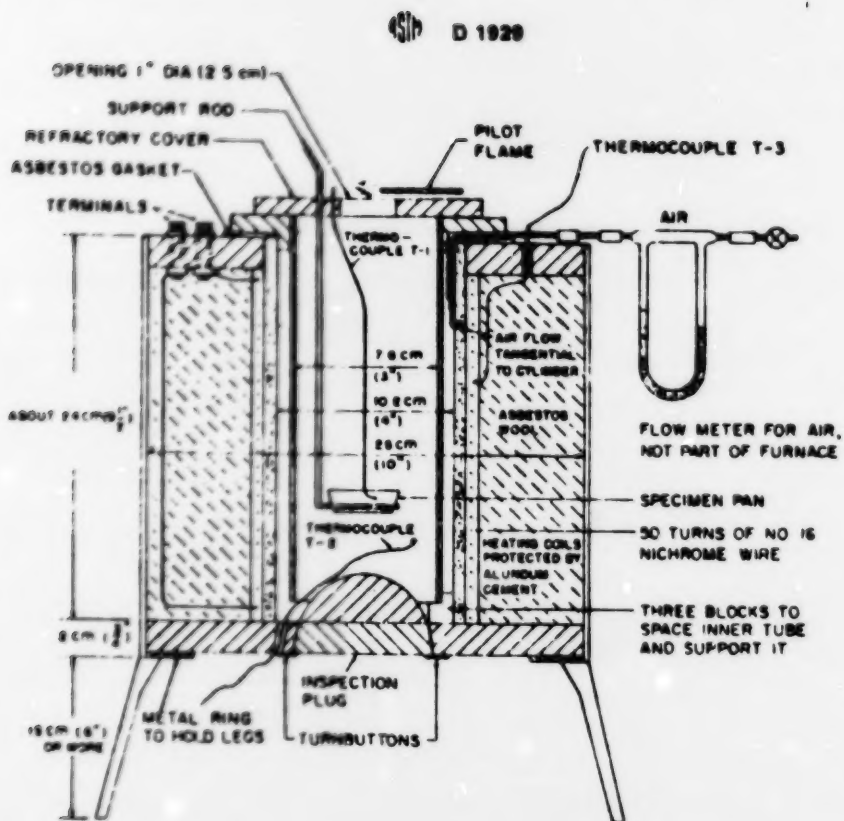


FIG. 1 Cross Section of Hot-Air Ignition Furnace Assembly.

Fig.2. Temperature Distribution - no crucible

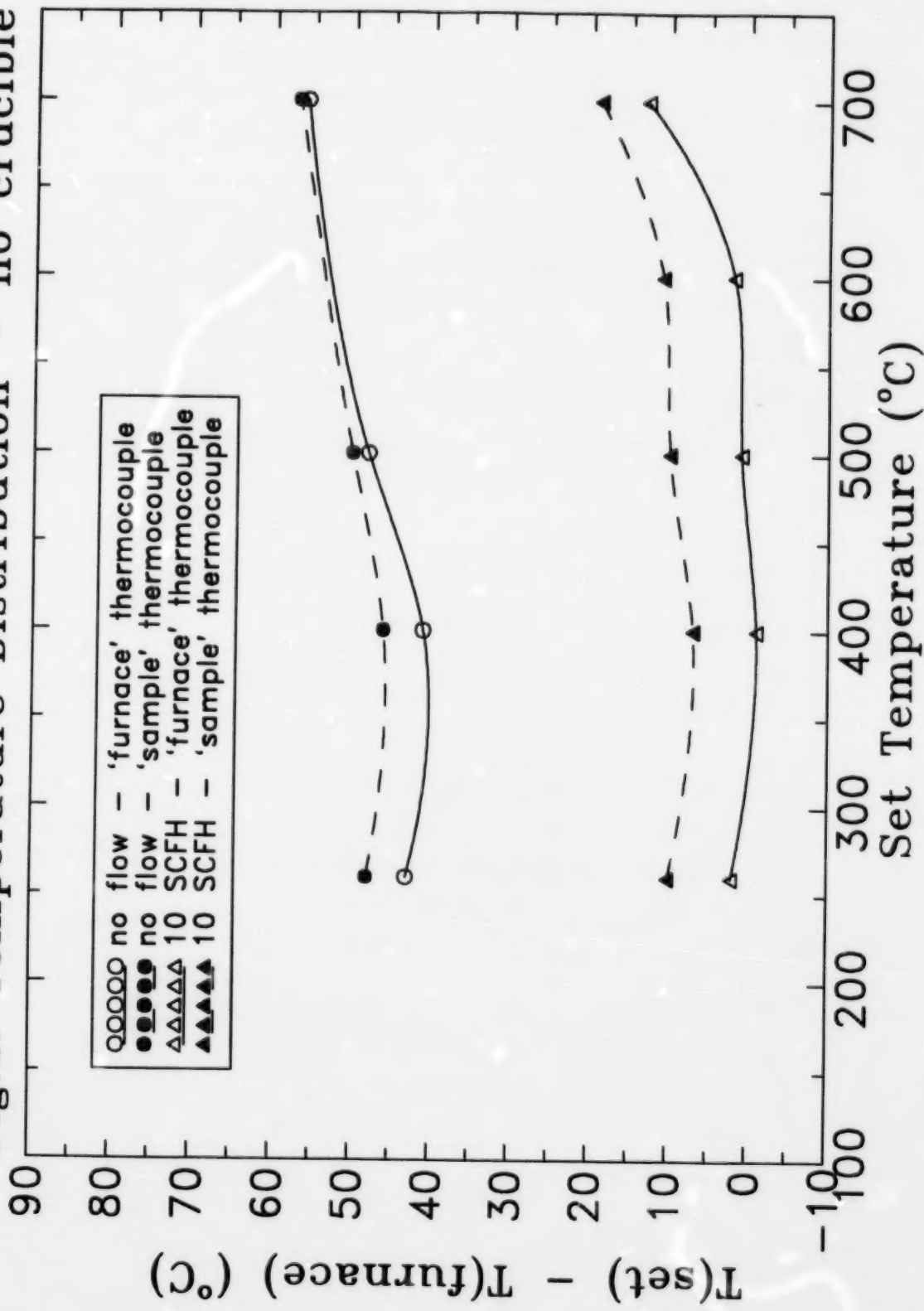


Fig.3. Temperature Distribution—with crucible

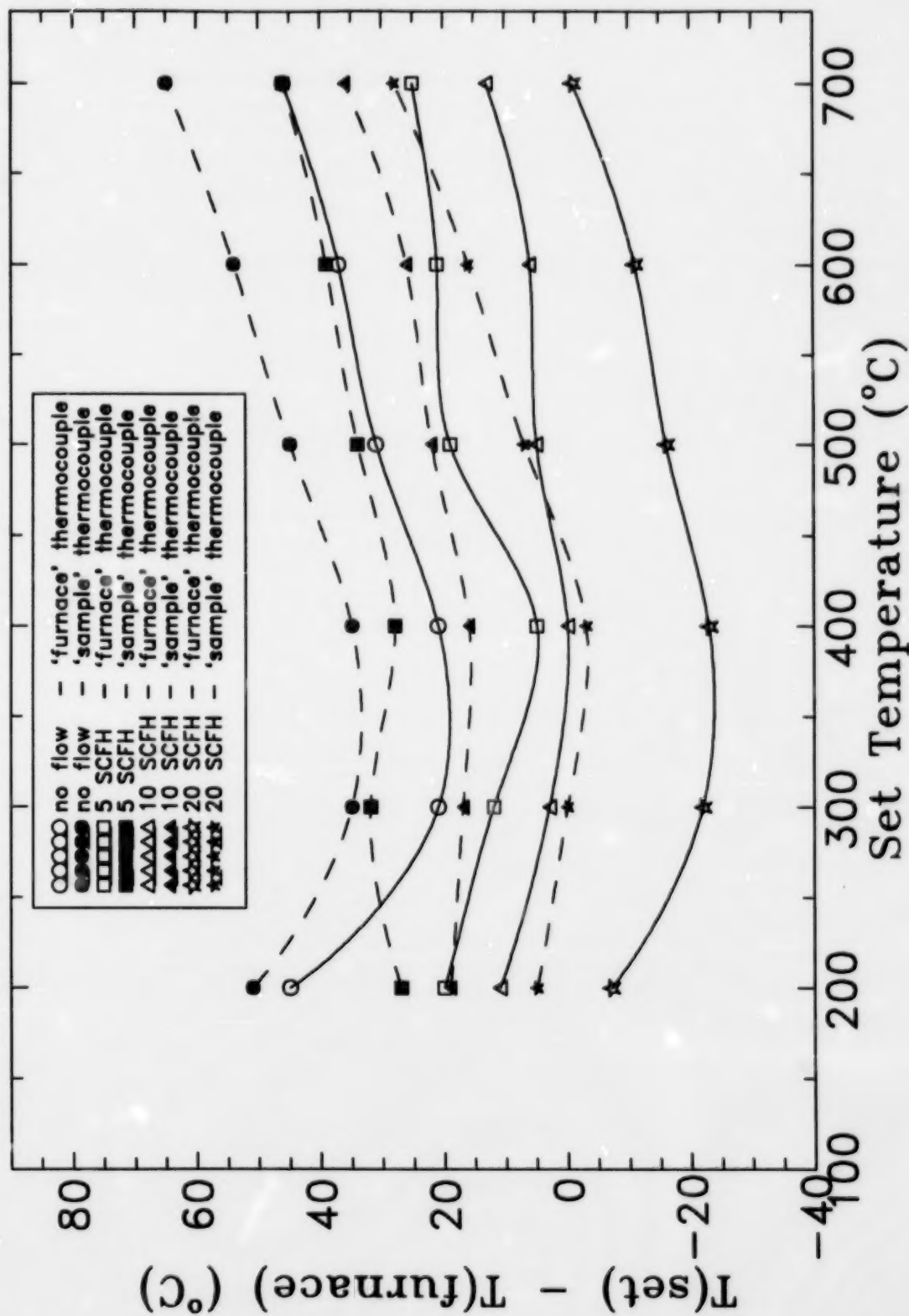


Fig.4. Pitts. Coal Dust: Effect of Layer Thickness

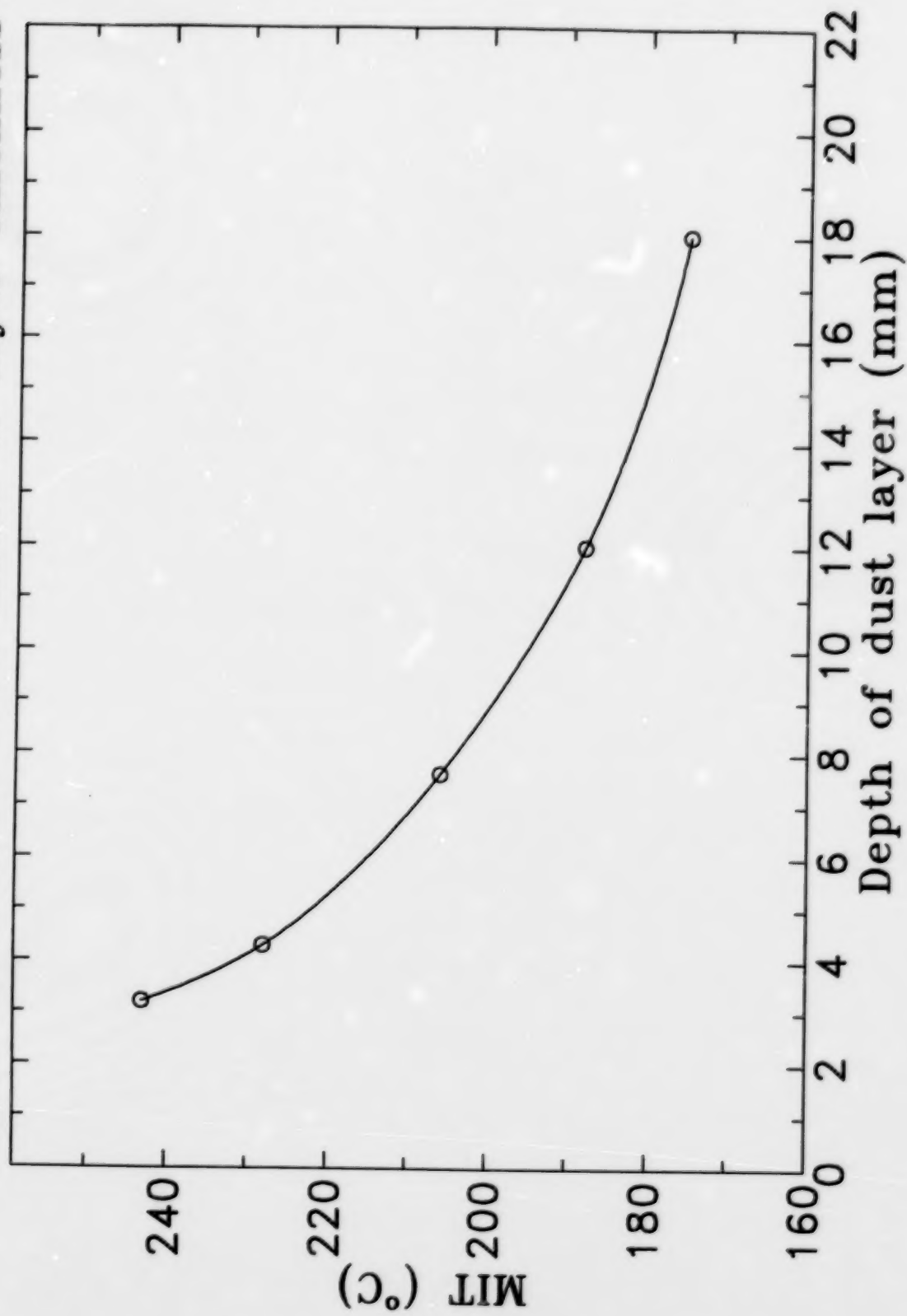


Fig.5. Pitts. Coal Dust: Effect of Sample Mass

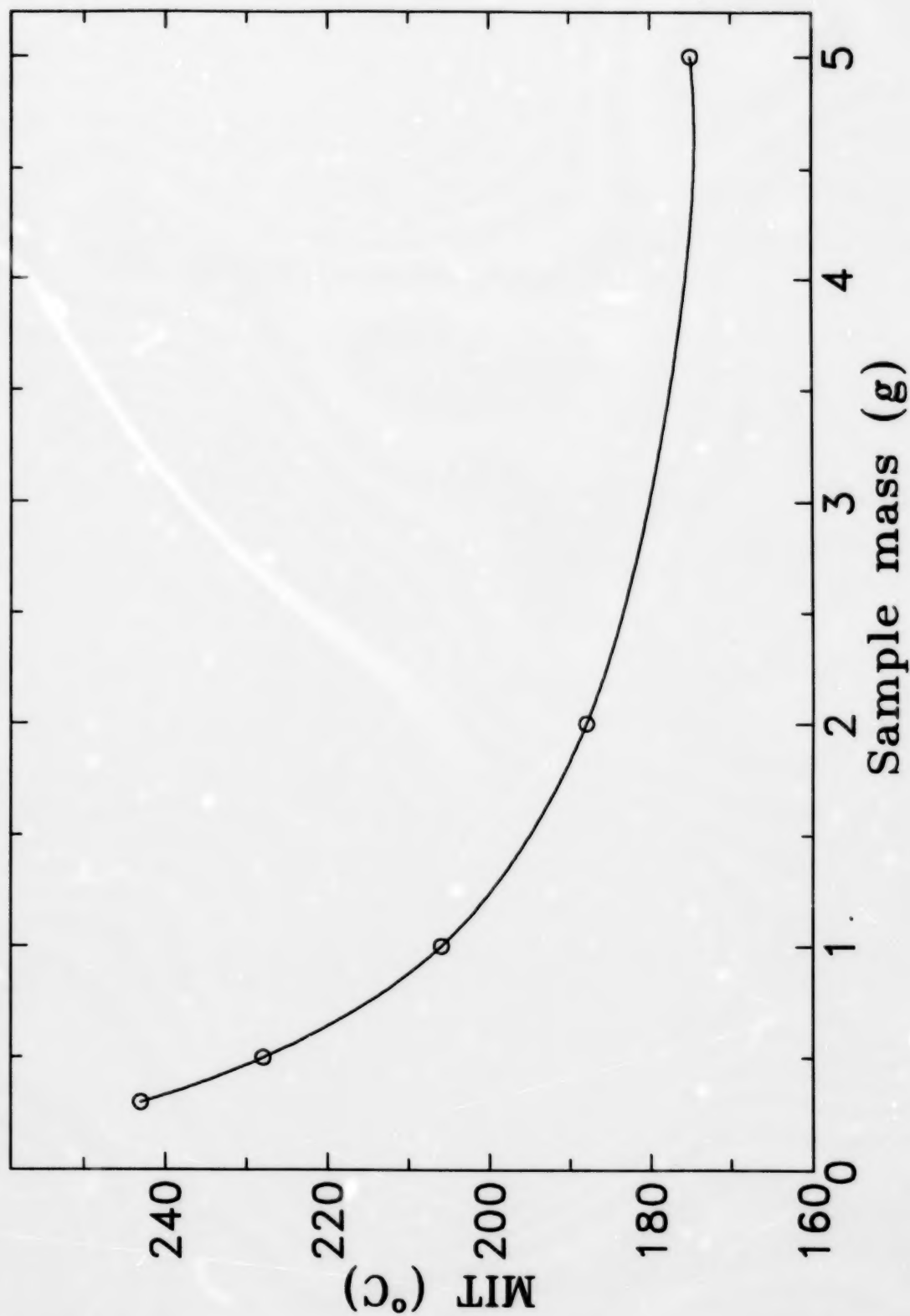


Fig.6. Pittsburgh Coal Dust: Flaming Combustion

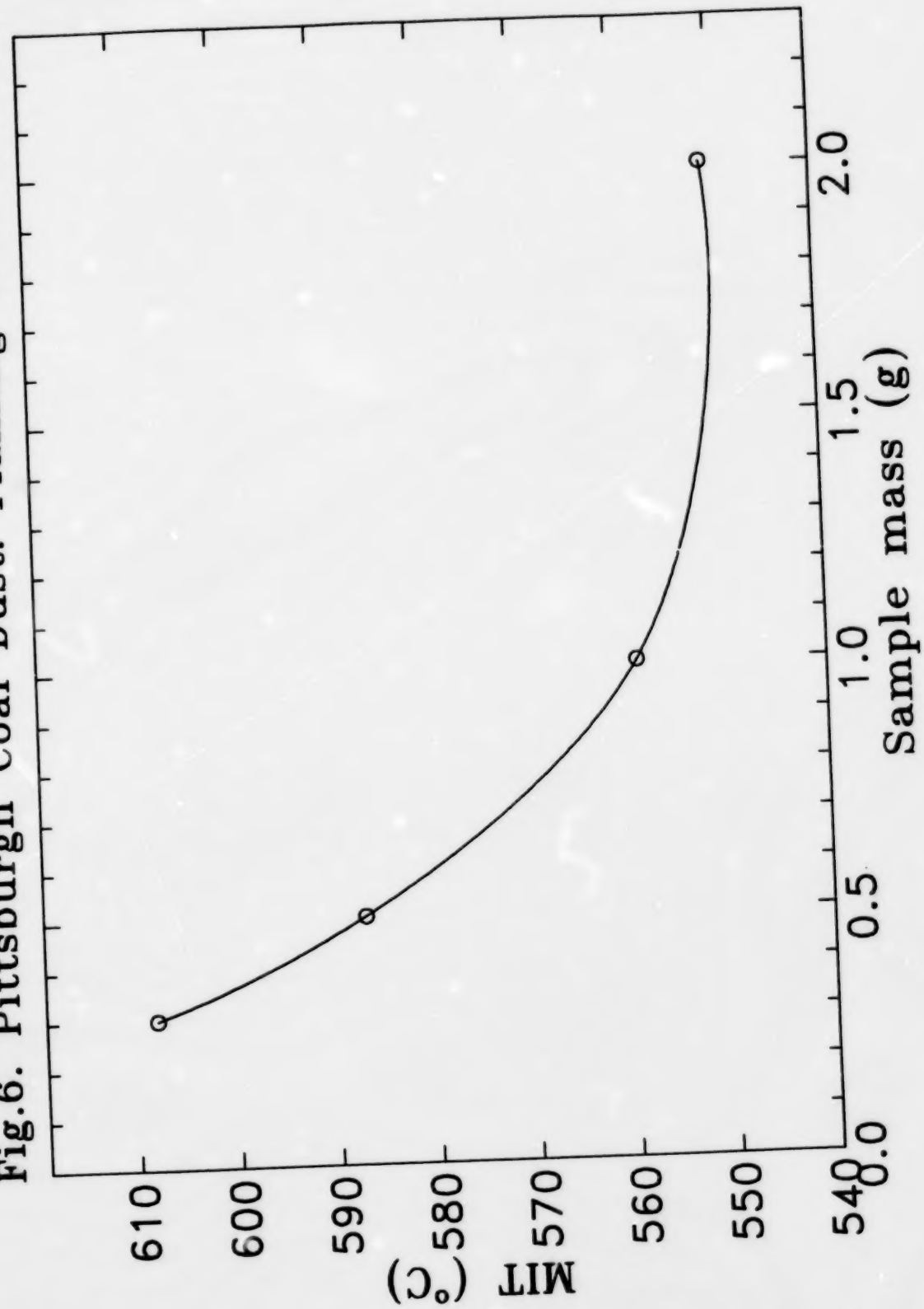


Fig.7. Lycopodium Dust: Effect of Layer Thickness

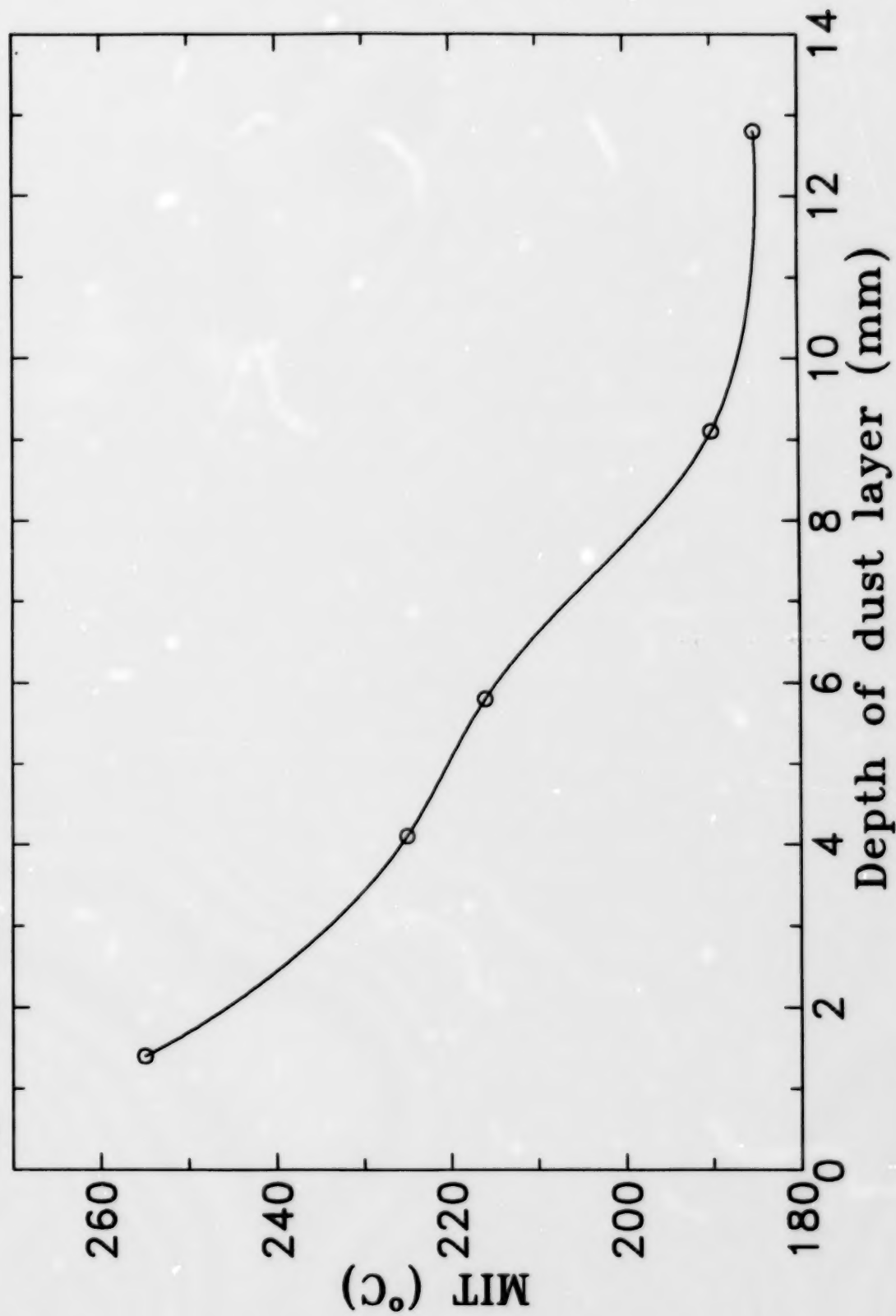


Fig.8. Lycopodium Dust: Effect of Sample Mass.

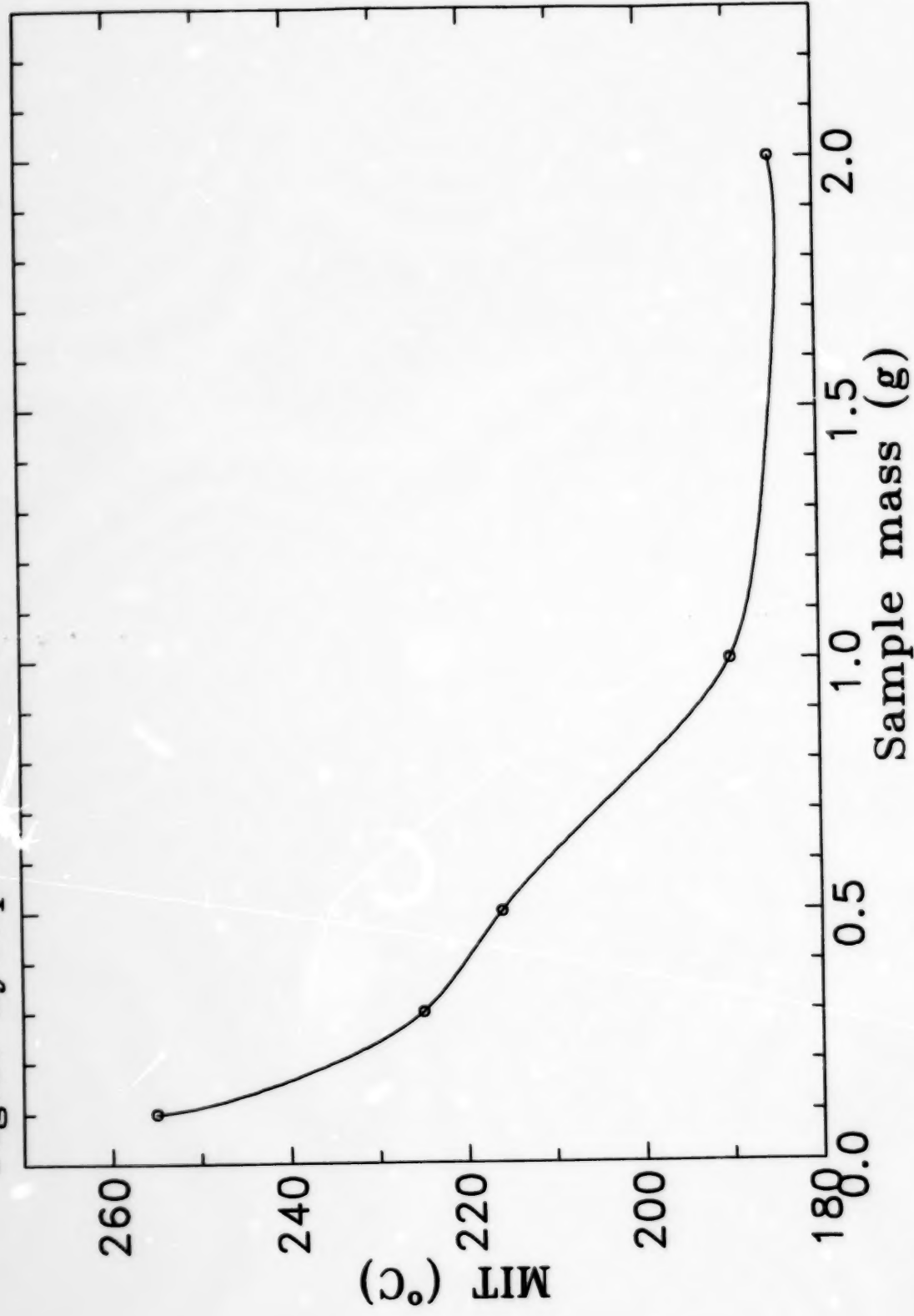
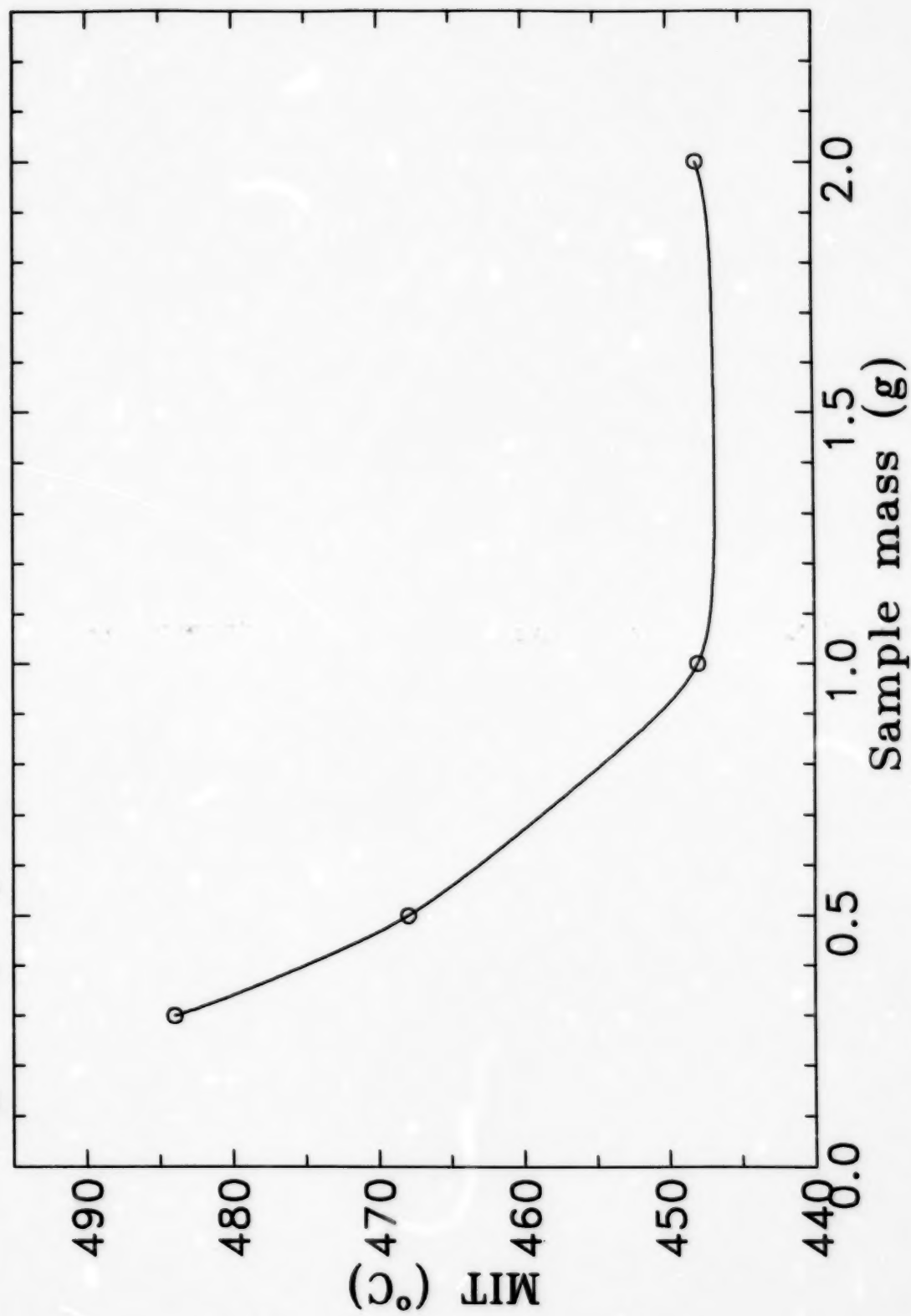


Fig.9. Lycopodium Dust: Flaming Combustion



APPENDIX

Safety Precautions

The main hazard of this test method is the high temperature involved. Care should be taken not to touch the furnace when it is hot and to handle the sample and furnace covers with tongs. It is also important not to place the sample container on any combustible surface when it is hot.

The second hazard is the noxious fumes that may be produced when the samples are heated. If the furnace is located inside the fume hood, and the windows of the fume hood mostly closed, then there should be no hazard from fumes. It is important to leave the sample container inside the fume hood until it has cooled.

The care in handling a particular sample will depend on the toxicity characteristics of that sample. The material safety data sheet should be read before carrying out tests.

The safety apparel required is: safety glasses with sideshields and lab coat.

Procedure

Set-up

1. Place the table containing the Setchkin apparatus in the fume hood. Lock the casters and ensure that the table is level. Place the operating console just to the right of the fume hood, and plug it in.
2. Place the ring stand such that the two thermocouples can be placed in the furnace through the hole in the split Pyrex cover, while being held by clamps on the stand.
3. Place the stand for the mirror so that the inner furnace can be observed from outside the fume hood.
4. Check that the "furnace" thermocouple is about 23 cm below the sample rod and the "sample" thermocouple is about 18 cm below the sample rod.

Note: Normally, it should not be necessary to make any adjustment.

5. Connect the thermocouple leads from the outer chamber of the furnace to station #5 on the operating console; the thermocouple lead from the "furnace" thermocouple to station #1 and the lead from the "sample" thermocouple to station #2. Connect the heating element and the Tygon air line to the furnace.
6. Turn on the main switch of the console.
7. Turn on the switch on the console for the air pump; adjust to 10 SCFH.
8. Open the data logger window and turn the switch on. The printer should start and print out a date, headings and temperatures. Check that the temperatures for stations 1 and 2 are

correct.

9. Press the "on-line" button on the printer, then press the "line feed" until the paper is at the top of a sheet. Then turn the data logger switch off and on again. That sets the top of page. Leave that switch on for the remainder of the tests. Use the "on-line" button for turning the printer off and on.

Rising temperature tests

1. Turn on the fume hood.
2. Weigh out the dust into a crucible (usually 2 g).
3. Put the crucible into the holder and hang it on the rod on the ring stand.
4. Use tongs to hold the top of the sample holder and place it carefully onto the sample rod.
5. Place the "sample" thermocouple into the furnace so that it is immersed in the dust.
6. Replace the split glass covers, so that there is no gap between them except for the centre hole. Close the fume hood window.
7. Turn the printer on.
8. Set the temperature dial to the desired maximum temperature.
9. Turn the heater switch on. The red light should go on indicating that the furnace is heating.
10. After the temperature has reached the set temperature, turn the heater off, with tongs slide one half of the Pyrex cover off, leaving it to rest on the top of the furnace, loosen the thermocouple clamp and raise the thermocouple and with tongs remove the sample holder and place it on the rod of the ring stand.
11. Check the temperature trace for sign for an inflection point. This can often be picked out easily by the temperature of the "sample" thermocouple rising above that of the "furnace" thermocouple. This will serve as the initial temperature in the constant temperature tests of the determination of $MIT_{\text{non-flaming}}$.

Note: If observations of reaction are observed, such as smoke or flames, then the heater can be turned off immediately and the temperature that these events begin at can be used as the starting point for the determination of $MIT_{\text{non-flaming}}$. For the determination of MIT_{flaming} , it is necessary to continue the test until a flame is observed.

Constant temperature tests: determination of $MIT_{\text{non-flaming}}$

1. Set the temperature dial to the starting temperature.
2. Place the "sample" thermocouple in the furnace and replace the covers.
3. After the furnace has reached the set-point, as indicated by the heater lights, turn the printer on and wait until the "furnace" thermocouple has stabilized.

4. Weigh out the required amount of dust (usually 2 g) into the crucible. Put the crucible in the sample holder.
5. With tongs, slide one-half of the cover off the inner tube, and hang the sample holder on the sample rod.
6. Put the "sample" thermocouple into the dust sample, clamp it into place, and with tongs slide the cover back into place.
7. Immediately turn on the stopwatch.
8. Watch the sample by means of the mirror. Note the time when smoke starts (if is observed).
9. If flames are observed, stop the stopwatch. The furnace temperature is greater than the MIT_{flaming} .
10. If no flames are observed, continue the test until the sample temperature has passed its maximum. If the maximum sample temperature is well above the furnace temperature, (a difference of more than 20°C), then the furnace temperature is above the $MIT_{\text{non-flaming}}$, the set temperature should be lowered 10°C for the next test. Otherwise, the set temperature should be raised 10°C for the next test.
11. Loosen the thermocouple clamp, and raise the thermocouple out of the dust. With tongs, slide the cover off the inner tube and remove the sample holder and place it on the stand rod until it cools.
12. Continue from step 2 until the MIT has been bracketed within 10°C .

Constant temperature tests: MIT_{flaming}

Same as for $MIT_{\text{non-flaming}}$ except that in step 10, the furnace temperature is below the MIT_{flaming} , therefore, the set temperature should be raised 10°C for the next test.